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Elevated Temperature In-situ Transmission Kikuchi Diffraction: a Powerful Tool for the Characterization of Ultra-thin Metal Films for Nanofabrication Applications

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The fabrication process of increasingly complex multi-material and multi-layer structures and devices in nanofabrication includes thermal treatments that might affect the nanostructure and stability of the films heavily. An important temperature-related problem for the application of such systems is solid-state dewetting¹, the agglomeration of the film to form islands or nanoparticles when heated to sufficiently high temperatures, which can have a harmful effect on the performances and time-stability of the fabricated devices.

The fundamental insight in the driving forces for changes in crystal structure, grain size and crystallographic texture of such systems due to thermal treatments requires the introduction of innovative techniques having nanoscale resolution and being able to operate in a wide range of temperatures.

In this study, we demonstrate the in-situ capabilities of transmission Kikuchi diffraction (TKD) for the analysis of ultra-thin Au films at high temperature. The dewetting of an Au thin film into Au nanoparticles upon heating is followed with orientation mapping in a temperature range between 20°C and 900°C. The evolution of grain size and film texture and the growth of holes in the film are tracked throughout the process with high resolution, accuracy and statistical significance. Several sources of influence on the quality and resolution of the acquired TKD maps are investigated: disturbance from infrared radiation, maximum measurable Au thickness, loss of crystalline order, thermal drift during heating, plasma cleaning of the sample and thickness variation of the film.

A quasi in-situ TKD mapping is also used to observe the positive impact on the stability of the Au nanostructure at elevated temperatures due to the presence of Ti and Cr transition metals used as adhesion layers. The results show that the continuity of the Au film is preserved up to 500°C using either of the adhesion layers (Fig. 1), but also how Cr and Ti have a different impact on the final Au film nanostructure.

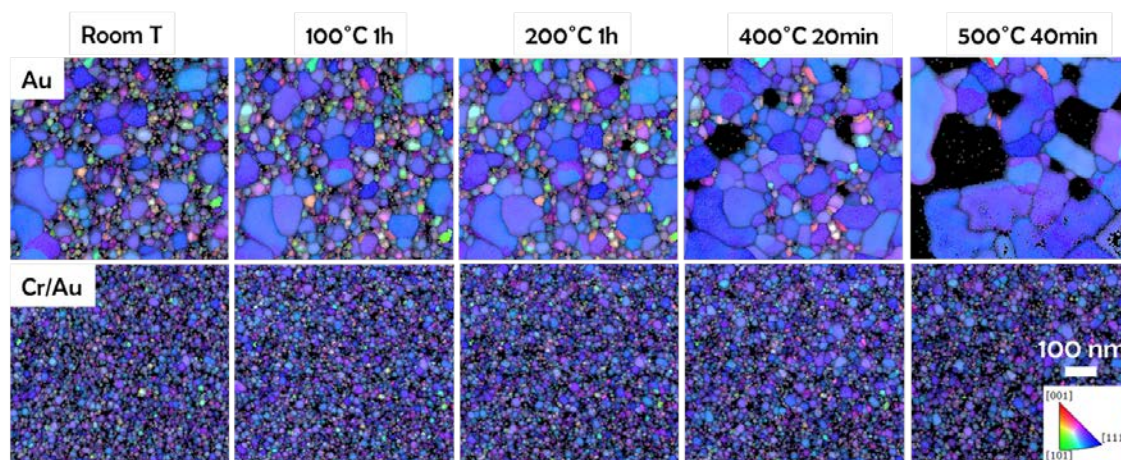


Figure 1: Inverse pole figure (IPF) quasi in-situ TKD maps showing the evolution of Au nanostructure without (top) and with Cr adhesion layer (bottom). The presence of the adhesion layer maintains the Au film continuous up to 500°C.

[1] C.V. Thompson, *Annu. Rev. Mater. Res.* **42**, 399–434 (2012).